**Contains No CBI**

RHÔNE-POULENC

**TOXICOLOGY DEPARTMENT**
P.O. BOX 12014, 2 T.W. ALEXANDER DRIVE
RESEARCH TRIANGLE PARK, NC 27709
(919) 549-2000 TELEFAX (919) 549-8525
INTERNATIONAL TELEX NUMBER 4999378–ANSWERBACK APC RTP

92 OCT 30 AM 8:29

October 29, 1992

(A)

VIA FEDERAL EXPRESS

8EHQ-92-12496

8892000106 81

INIT

Document Processing Center (TS-790)
Office of Toxic Substances
US Environmental Protection Agency
401 M Street, SW
Washington, DC 20460

Attn: Section 8(e) Coordinator (CAP Agreement)

RE: Report Submitted Pursuant to the TSCA Section 8(e) Compliance Audit Program

CAP ID No.: 8ECAP - 0004

Dear Sir/Madam:

On behalf of Rhône-Poulenc Inc. (RPI, CN 5266, Princeton, NJ 08543-5266) and its subsidiary Rhône-Poulenc Ag Company (RPAC), the following information is being submitted to the Environmental Protection Agency (EPA) pursuant to the Toxic Substances Control Act (TSCA) Section 8(e) Compliance Audit Program and the Agreement for a TSCA Section 8(e) Compliance Audit Program (CAP Agreement) executed by RPI and EPA.

The enclosed information on 1,2,3,4-tetrahydronaphthalene (CAS number 119-64-2), naphthalene (CAS number 91-20-3), biphenyl (CAS number 92-52-4), and 1,1'-oxybis-benzene (CAS number 101-84-8). No claims of confidentiality are made for this submission.

This report is being submitted under Section 8(e) because measurable amounts of the afore-mentioned chemicals were found in fish following the release into a river of some in-process material from our SEVIN® complex. (SEVIN is a registered pesticide.) This release was due to an explosion that occurred at the plant, and a large fish kill was reported the day after the release. Four samples of fish were submitted for analysis of specific chemicals. Two of the fish samples were taken upstream from the manufacturing plant while the other two samples were taken near the plant river bank. For all four chemicals, residues were higher in the fish taken near the plant compared to those taken from upstream. Information on the concentration of these chemicals in the river and the amount of the release is not available. Therefore, no correlations between the amount released and the concentrations in the fish can be made.

No previous TSCA Section 8(e) notices have been submitted on these chemicals. In total, RPI is submitting three copies of the report and this cover letter: an original and two copies. Further questions regarding this submission may be directed to the undersigned at 919-549-2222.

Sincerely,

Glenn S. Simon

8/2/95

Glenn S. Simon, PhD, DABT
Director of Toxicology

# RHÔNE-POULENC AG COMPANY

INSTITUTE PLANT

## OFFICE MEMORANDUM

| | | | |
|---|---|---|---|
| TO: | F. L. Boggs | DATE: | January 23, 1989 |
| FROM: | D. F. Holley | DEPT: | Environmental Protection |
| CC: | R. L. McNeer | SUBJECT: | Fish Sample Results from Wright State University |

The data package has been received from Wright State University containing results from the fish samples submitted after the ethylene oxide incident of August, 1988. As you know, four samples were submitted for analyses. Two of the four samples - 1) Blue Gill and Bass and; 2) Catfish were taken upstream of the Plant near the Dunbar Bridge. The other two samples were taken near the Plant river bank and were dead when obtained. The results of all four samples are included in Attachment I.

As is apparent, there is a distinct difference between the two sets of samples. Those taken at the Plant contain significant amounts of the four major organics lost to the Kanawha River, presumably from the SEVIN™ Unit.

Attached for your review is a copy of the letter from Dr. Tiernan (Wright State) and several other documents contained in the data package. One important observation is that the minimum detectable quantity for measuring these organics in fish is 0.0025 ppm. The entire data package will be in the Central Files along with a copy of the final EPD report on the incident.

If there are any questions or other actions you would like taken, please let me know.

Very truly yours,

Diana Holley

D. F. Holley



0855Q/tlq

## Attachment I
(ug/g-ppm)

| | Blue Gill/Bass<br>Upstream - Dunbar Bridge | Catfish<br>Upstream - Dunbar Bridge |
|---|---|---|
| Tetralin | ND | 0.021 |
| Naphthalene | ND | 0.013 |
| 1,1-Biphenyl | ND | 0.042 |
| 1,1-Oxydisbenzene | 0.023 | 0.102 |

| | Minnows<br>Plant River Bank | Bass<br>Plant River Bank |
|---|---|---|
| Tetralin | 73.6 | 22.7 |
| Naphthalene | 15.7 | 4.37 |
| 1,1-Biphenyl | 3.6 | 0.86 |
| 1,1-Oxydisbenzene | 77.3 | 2.04 |

0855Q/2
/tlq

WRIGHT STATE

Wright State University
Dayton, Ohio 45435

January 12, 1989



Ms. Diana Holley
Rhone-Poulenc AG Company
Building 330
Route 25
Institute, West Virginia 25112

Dear Ms. Holley:

Presented herewith is the report of the results of analyses accomplished by our laboratory to characterize major chemical residues in fish samples submitted by Rhone-Poulenc. These analyses were accomplished under Rhone-Poulenc AG Company Purchase Order No. 0512-105704. Since these analyses were requested in connection with an accidental release of chemicals to the river in which the fish were collected, the major target analytes were the compounds known to have been released. As indicated in telephone discussions with you and in your letter of August 17, 1988 to me, these compounds included ethylbenzene, tetralin (1,2,3,4-tetrahydronaphthalene), naphthalene, 1,1'-biphenyl, 1,1'-oxybisbenzene, substituted indones and substituted tetralins. Pure standards of all these compounds except the last two were available in our laboratory at the time this project was initiated, but the latter two compounds could not be obtained in a reasonable time period, and consequently, rigorous quantitative analyses were accomplished only for the other five compounds indicated. As will be discussed below, however, the analytical procedure applied here incorporates a qualitative GC-MS screening of samples for any and all chemical residues which may be present.

The fish samples received from Rhone-Poulenc for analyses are described in the Sample Receipt Documentation shown in Attachment A to this report. As shown therein, four types of fish samples were provided for analyzes.

At the outset of this project, it was recognized that there is no extant analytical methodology which is intended to quantitatively measure in a single analytical procedure the specific target analytes which are of interest here in fish tissue. However, it was thought that a procedure jointly developed by our laboratory and the U.S. EPA/Environmental Research Laboratory (Duluth) for multi-residue determinations of a wide variety of zenobiotic compounds in fish might be applicable, with appropriate modifications, for this purpose. An analytical protocol which describes these procedures in some detail in presented in Attachment B to this report. It should be realized that the methods described therein are still being refined somewhat, and are quite new in terms of demonstrated

application. However, our laboratory has previously tested these procedures in a survey of some 200 fish samples in connection with a Bioaccumulation Study being conducted by the U.S. EPA, and the EPA/ERL(Duluth) facility has used this method to characterize a similar number of fish samples. As can be seen from this protocol in Attachment B, the method entails grinding and homogenizing the fish samples, Soxhlet extraction of the ground sample, Gel Permeation Chromatographic fractionation of the extract to remove the bulk of the fish lipid and concentrate the target analytes in an appropriate fraction, silica gel cleanup to remove additional residual lipid, and finally, analysis of the processed extract using GC-MS. Since some of the target analytes for the present study are not included in the list of target analytes shown in the Analytical Protocol in Attachment B, it was necessary to verify that the compounds of interest here would indeed be recovered and measured using these procedures. Accordingly, a solution of the five target analyte species was prepared and the applicability of the methods was briefly assessed. It appeared from these initial experiments that these analytes could be recovered using the procedures described in the protocol, and therefore we proceeded to implement these methods for the fish samples submitted by Rhone-Poulenc.

It will be seen from the protocol presented in Attachment B that the method utilizes three deuterated internal standards, $d_{10}$-biphenyl, $d_{10}$-phenanthrene and $d_{12}$-chrysene, which are used as the basis for quantitating the target analytes. The method also utilizes three surrogates, iodobenzene, iodonaphthalene, and 4,4'-diiodobiphenyl, which exhibit GC retention times spanning approximately those of the target analytes. The recoveries achieved for these surrogates provide an indication of the overall efficacy of the method. The method also provides for comparison of the mass spectra of the target analytes, internal standards, and surrogates with those of authentic standards of those compounds which are resident in the Mass Spectral Library stored in the MS data system. A good comparison or "fit" (the best fit corresponding to a "Fit" factor of 1.00) provides a confirmation of the identification made, and in conjunction with GC retention time comparisons of the unknown peaks and authentic standards of the target analytes, yields quite specific identifications. Non-target analyte peaks which are detected in the TIC chromatograms for the GC-MS analyses are also subjected to library searches in an effort to qualitatively identify these components.

In the present analyses, solutions of the five target analyte compounds and the internal standards and surrogates mentioned above were prepared in a series of different concentrations ranging from 2 to 40 ng/µl. These solutions were used to establish calibration plots over an appropriate range of concentration for the target analytes. These plots are shown in Attachment F to this report and exhibit reasonably good linearity

over the concentration ranges indicated.

Additional details of the analyses are provided in the Intralaboratory Sample Tracking Form shown in Attachment C to this report. This form, which accompanies the samples as they proceed through the several stages of analysis in the laboratory, shows the weights of the aliquots of ground/homogenized fish which were analyzed, the quantities of internal standards added prior to analyses, the dates of sample preparation and GC-MS analyses, the percent lipid in each fish sample, and the extract final volume. Also indicated on this form are the names of the principal analysts and reference citations to the laboratory notebooks where full details of the analyses are recorded.

The results of the analyses are summarized in a set of tables presented in Attachment D to this report. The results for each sample, beginning with a laboratory blank, are shown on a separate page. The sample to which each page in this attachment is relevant is shown at the top of the page, under "Customer ID". The quantitative results for the five target analytes mentioned earlier are shown in the last section of each page in Attachment D, under "Conc."(ng/g)". In cases where the analyte was not detected, the minimum detectable concentration is indicated under "MDQ (ng/g).

It can be seen from the data for the Lab Blank which are shown in Attachment D that ethylbenzene is present in the blank at a concentration of 13.1 ng/g (ppb). This was determined to present in the Reagent Grade Toluene which was used to extract the fish. Since this level exceeds or essentially equals (within the experimental error of measurement) the concentrations of ethylbenzene reported in the fish samples, as shown on the other data pages in Attachment D, it is clear that there are no significant levels of ethylbenzene in the fish, and that the observed concentrations of this compound in the fish are accounted for by the laboratory solvent background. As also seen from the results summarized in Attachment D, none of the other four target analytes were detected in the Lab Blank, but one or more of these compounds were detected and quantitated in all four of the fish samples analyzed, at varying concentrations. The "Blue Gill and Bass" sample and the "Catfish" sample submitted by you exhibit lower (or non-detectable levels) of the analytes, while the "Minnows" sample and the "Bass" sample contain relatively high concentrations of all four analytes in question. These identifications are quite reliable in terms of "fit" of the mass spectra for the relevant GC peaks to the corresponding spectra of the standards.

It will also be observed from the data in Attachment D that the indicated recoveries of one or more of the surrogate compounds are low. However, this is not actually due to failure to recover these surrogates, but to the failure of the MS library comparison to identify the surrogate compound within the

specified "fit" limits. This is due to extensive interferences to the spectra arising from other compounds in the sample extracts which populate the same mass spectral peaks as those used for indicators of the surrogates. In such cases, the MS system software automatically shows zero recovery of the surrogate. Therefore, the surrogate data presented in the tables in Attachment D are not reliable indicators of the efficiency of the method. In contrast to the behavior of the surrogates, all of the internal standards used in these analyses were readily identified by the data reduction procedures, and provided reliable bases for quantitating the target analytes.

Additional analytical results are provided in Attachment E to this report. Here, a series of data packages are provided, one for the lab blank and for each of the four fish samples analyzed, which include a front summary page, followed by a Total Ion Current Mass Chromatogram (TIC), (analogous to a Gas Chromatogram), and a table which summarizes the areas of the major non-target analyte peaks detected in the TIC. Following the latter table is a listing of the compounds which correspond to the ten best "fit" matches resulting from the MS library search accomplished for the first major non-target analyte peak in the TIC. This is followed by a graphical (bar graph) display of the mass spectra for the three best "fit" identifications, and a second graphical display of the background mass spectrum, the spectrum of the best "fit" compound identified and the difference between these two spectra. A similar set of compound listings and mass spectra is then given for each of the other major non-target analyte peaks detected in the TIC. A review of the mass spectral search results presented for the samples in Attachment E indicates that most of the qualitative identifications made are not meaningful because the "fit" values are too low. Generally, "fit" values less than about 0.6 are suspect.

This concludes our analyses of the fish samples submitted by Rhone-Poulenc. If you have any further questions concerning the results or the report, don't hesitate to contact us. Under separate cover, our financial services office will submit an invoice for these analyses. We appreciate the opportunity to work with Rhone-Poulenc on this important problem.

Sincerely,

Thomas O. Tiernan, Ph.D.
Professor of Chemistry, and
Director of the Contaminant
Research Program

Attachments

# *Triage of 8(e) Submissions*

Date sent to triage: 5/25/96 NON-CAP (CAP)

Submission number: 12496A TSCA Inventory: (Y) N D

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Study type (circle appropriate):

Group 1 - Gordon Cash (1 copy total)

ECO (AQUATO)

Group 2 - Ernie Falke (1 copy total)

ATOX SBTOX SEN w/NEUR

Group 3 -HERD (1 copy each)

STOX CTOX EPI RTOX GTOX

STOX/ONCO CTOX/ONCO IMMUNO CYTO NEUR

Other (FATE, EXPO, MET, etc.): EXPO

---

Notes:

☒ This is the **original** 8(e) submission; refile after triage evaluation.

☐ This **original** submission has been **split**; rejoin after triage evaluation.

☐ Other:

**Photocopies Needed for Triage Evaluation**

**entire document:** (0) 1 2 3

**front section and CECATS:** (0) 1 2 3

**Initials:** JD **Date:** 5/23/96

CECATS\TRIAGE TRACKING DBASE ENTRY FORM

CECATS DATA:
Submission # 8EHQ- 1092-12496 SEQ. A

TYPE: INT. SUPP FLWP

SUBMITTER NAME: Rhone-Poulenc Inc.

SUB. DATE: 10/29/92 OTS DATE: 10/30/92 CSRAD DATE: 08/02/95

INFORMATION REQUESTED: FLWP DATE:
0501 NO INFO REQUESTED
0502 INFO REQUESTED (TECH)
0503 INFO REQUESTED (VOL ACTIONS)
0504 INFO REQUESTED (REPORTING RATIONALE)

DISPOSITION:
0639 REFER TO CHEMICAL SCREENING
0678 CAP NOTICE

VOLUNTARY ACTIONS:
0401 NO ACTION REPORTED
0402 STUDIES PLANNED/UNDERWAY
0403 NOTIFICATION OF WORKERS/USERS
0404 LABEL/MSDS CHANGES
0405 PROCESS/HANDLING CHANGES
0406 APP./USE DISCONTINUED
0407 PRODUCTION DISCONTINUED
0408 CONFIDENTIAL

CHEMICAL NAME: CAS#

Selvin → none 119-64-2

91-20-3

101-84-8

| INFORMATION TYPE: | | P F C |
|---|---|---|
| 0201 | ONCO (HUMAN) | 01 02 04 |
| 0202 | ONCO (ANIMAL) | 01 02 04 |
| 0203 | CELL TRANS (IN VITRO) | 01 02 04 |
| 0204 | MUTA (IN VITRO) | 01 02 04 |
| 0205 | MUTA (IN VIVO) | 01 02 04 |
| 0206 | REPRO/TERATO (HUMAN) | 01 02 04 |
| 0207 | REPRO/TERATO (ANIMAL) | 01 02 04 |
| 0208 | NEURO (HUMAN) | 01 02 04 |
| 0209 | NEURO (ANIMAL) | 01 02 04 |
| 0210 | ACUTE TOX. (HUMAN) | 01 02 04 |
| 0211 | CHR. TOX. (HUMAN) | 01 02 04 |
| 0212 | ACUTE TOX. (ANIMAL) | 01 02 04 |
| 0213 | SUB ACUTE TOX (ANIMAL) | 01 02 04 |
| 0214 | SUB CHRONIC TOX (ANIMAL) | 01 02 04 |
| 0215 | CHRONIC TOX (ANIMAL) | 01 02 04 |

| INFORMATION TYPE: | | P F C |
|---|---|---|
| 0216 | EPI/CLIN | 01 02 04 |
| 0217 | HUMAN EXPOS (PROD CONTAM) | 01 02 04 |
| 0218 | HUMAN EXPOS (ACCIDENTAL) | 01 02 04 |
| 0219 | HUMAN EXPOS (MONITORING) | 01 02 04 |
| 0220 | ECO/AQUA TOX | 01 02 04 |
| 0221 | ENV. OCCC/REL/FATE | 01 02 04 |
| 0222 | EMER INCI OF ENV CONTAM | 01 02 04 |
| 0223 | RESPONSE REQEST DELAY | 01 02 04 |
| 0224 | PROD/COMP/CHEM ID | 01 02 04 |
| 0225 | REPORTING RATIONALE | 01 02 04 |
| 0226 | CONFIDENTIAL | 01 02 04 |
| 0227 | ALLERG (HUMAN) | 01 02 04 |
| 0228 | ALLERG (ANIMAL) | 01 02 04 |
| 0239 | METAB/PHARMACO (ANIMAL) | 01 02 04 |
| 0240 | METAB/PHARMACO (HUMAN) | 01 02 04 |

| INFORMATION TYPE: | | P F C |
|---|---|---|
| 0241 | IMMUNO (ANIMAL) | 01 02 04 |
| 0242 | IMMUNO (HUMAN) | 01 02 04 |
| 0243 | CHEM/PHYS PROP | 01 02 04 |
| 0244 | CLASTO (IN VITRO) | 01 02 04 |
| 0245 | CLASTO (ANIMAL) | 01 02 04 |
| 0246 | CLASTO (HUMAN) | 01 02 04 |
| 0247 | DNA DAM/REPAIR | 01 02 04 |
| 0248 | PROD/USE/PROC | 01 02 04 |
| 0251 | MSDS | 01 02 04 |
| 0299 | OTHER | 01 02 04 |

TRIAGE DATA: NON-CBI INVENTORY
YES
CAS SR NO
[illegible]

ONGOING REVIEW
YES (DROP/REFER)
NO (CONTINUE)
[illegible]

SPECIES
Fish

TOXICOLOGICAL CONCERN:
LOW
MED
HIGH

USE:
Pesticide

PRODUCTION:

[illegible]